

Virginia Division of Consolidated Laboratory Services

TKN Macro-Kjeldahl Digestion and Distillation					SM 4500-N_{org} B – 1997 (2011)	
<i>ADDITIONAL QC REQUIREMENTS FOR THIS METHOD: Certified or Accredited laboratories using this method are assessed to applicable requirements of SM 1020 and SM 4020.</i>						
Facility Name: _____ VELAP ID _____						
Assessor Name: _____ Analyst Name: _____ Inspection Date _____						
Records Examined: SOP Number/ Revision/ Date _____ Analyst: _____						
Sample ID: _____ Date of Sample Preparation: _____ Date of Analysis: _____						

Relevant Aspect of Standards	Method Reference	Y	N	N/A	Comments
Are sample volumes selected according to the expected organic nitrogen in samples? [0- 1 mg/L= 500 mL sample 1-10 mg/L=250 mL sample] [10- 20 mg/L=100 mL sample 20-50 mg/L= 50 mL sample] [50-100 mg/L= 25 mL sample]	4500-N _{org} B.4.a				
Are wet portions of sludge or sediment samples used for kjeldahl nitrogen determination? [NOTE: Measure dry weight of sample on a separate portion]	4500-N _{org} B.4.b				
Is ammonia removed by adding 25 mL borate buffer, adjusting pH to 9.5 with 6N NaOH, and boiling?	4500-N _{org} B.4.b				
Are boiling beads or chips added to each flask?	4500-N _{org} B.4.b				
For Digestion:					
Is the heating device preheated for 10 minutes if gas or 30 minutes if electric?	4500-N _{org} B.2.a				
Is 50 mL digestion reagent added to each flask, with a few additional boiling beads or chips, and mixed?	4500-N _{org} B.4.c				
Are samples boiled briskly until volume is greatly reduced and copious white fumes are observed? [NOTE: Fumes may be dark for samples high in organic matter]	4500-N _{org} B.4.c				
After fumes are observed, are samples digested for an additional 30 minutes? [NOTE: colored or turbid samples will become transparent and pale green]	4500-N _{org} B.4.c				

Notes/Comments:

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Relevant Aspect of Standards	Method Reference	Y	N	N/A	Comments
Following digestion, are samples cooled, diluted to 300 mL with deionized water, and mixed?	4500-N _{org} B.4.c				
Is 50 mL hydroxide-thiosulfate reagent carefully added to each cooled sample to form an alkaline layer at the bottom of the flask (pH > 11.0)?	4500-N _{org} B.4.c				
For Distillation: (Not required if use block digestion)	40CFR136.3 Footer 20				
Is each flask connected to the distillation apparatus then swirled to mix?	4500-N _{org} B.4.c				
Are samples distilled with the vertical condenser's outlet tip submerged well below the surface of the absorbent solution?	4500-N _{org} B.4.d				
Is 200 mL of distillate collected?	4500-N _{org} B.4.d				
Is the condenser temperature not allowed to rise above 29° C?	4500-N _{org} B.4.d				
Is the appropriate absorbent solution used for the final measurement technique? Titrimetric determination: 50 mL indicating boric acid solution Phenate or ISE determination : 50 mL 0.04N H ₂ SO ₄	4500-N _{org} B.4.d				
Are blanks and standards carried through all steps of the procedure?	4500-N _{org} B.4.f				

Notes/Comments: